

**PATENT APPLICATION**

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the Application of

Akinori KOUKITSU et al.

Group Art Unit: 1722

Application No.: 10/509,177

Examiner: M. SONG

Filed: September 27, 2004

Docket No.: 121213

For: VAPOR PHASE GROWTH METHOD FOR A1-CONTAINING III-V GROUP  
COMPOUND SEMICONDUCTOR, AND METHOD AND DEVICE FOR  
PRODUCING A1-CONTAINING III-V GROUP COMPOUND  
SEMICONDUCTOR

**DECLARATION UNDER 37 C.F.R. §1.132**

I, Koukito Akinori, a citizen of Japan, hereby declare and state:

1. I have a doctorate degree in Semiconductor Electronics which was conferred upon me by Tohoku University in Miyagi, Japan in 1981.

2. I have been employed by Tokyo University of Agriculture and Technology TLO Co., Ltd. since 1977 and I have had a total of 32 years of work and research experience in Semiconductor Electronics.

3. I am a named inventor in the above-captioned patent application.

4. I have a professional relationship with Tokyo University of Agriculture and Technology TLO Co., Ltd., assignee of the above-identified patent application. In the course of that professional relationship, I received compensation directly from Tokyo University of Agriculture and Technology TLO Co., Ltd. for my work relating to semiconductor electronics. I am being compensated for my work in connection with this Declaration.

5. I and/or those under my direct supervision and control have conducted the following experiments.

The data from the experiments demonstrates the criticality of reacting a solid Al with a halogenated hydrogen at a temperature of 700°C or below in a first reaction zone of the reaction chamber, which is made solely of quartz, during the first step of the reaction. By reacting a solid Al with a halogenated hydrogen at a temperature of 700°C or below in the first reaction zone, an Al component such as  $\text{AlCl}_3$  formed in the first reaction zone is transported into the second reaction zone and prevents the quartz reaction tube of the second reaction zone from being corroded, damaged or discolored by the Al component therein.

The experimental data includes data obtained from evaluating a depth profile of the AlN layer at the second reaction zone of the reaction chamber (quartz reaction tube) via secondary ion mass spectroscopy (SIMS) as shown in Graphs 1 and 2 of the present declaration. The experimental data also includes data obtained from evaluating an absorption spectra for the AlN layer at the second reaction zone of the reaction chamber as shown in Graphs 4 and 5 of the present declaration.

Graphs 1 and 2 illustrate SIMS depth profiles of the AlN layer for the examples disclosed in FIGS. 4 and 5 of the present application. As set forth in the present application, the temperature of the Al material area (first reaction zone of the reaction chamber) of the comparative example disclosed in FIG. 4 was set to 850°C and  $\text{AlCl}$  gas formed in Al material area was transported from the Al material area into the crystallization area (second reaction zone of the reaction chamber). In the example disclosed in FIG. 5 of the present application, the temperature of the Al material area (first reaction zone of the reaction chamber) was set to 650°C and substantially no  $\text{AlCl}$  gas was generated.

By comparing the SIMS depth profiles of the AlN layer of the second reaction zone for the comparative example and example of FIGS. 4 and 5, respectively, of the present

application as set forth in Graphs 1 and 2, the intensity of [C] and [O] in the AlN layer are substantially the same as or similar to each other. However, the intensity of [Si] for the comparative example disclosed in FIG. 4 of the present application (temperature of Al material area set to 850°C) as shown in Graph 1 is over 100 times higher than the intensity of [Si] for the example disclosed in FIG. 5 of the present application (temperature of Al material area set to 650°C). This high intensity of [Si] for the example disclosed in FIG. 5 is caused by deoxidizing and corroding of the quartz reaction tube by AlCl gas, the Al component, present at the second reaction zone. As a result of the reacting the solid Al with a halogenated hydrogen at a temperature of 850°C (example of FIG. 4 of the present application) in a first reaction zone, the quartz reaction tube at the second reaction zone is deoxidized and corroded by the Al gas and Si is emitted.

Graphs 3 and 4 disclose absorption spectra of AlN layer at the second reaction zone when the Al material area (first reaction zone) is set to 600°C and 850°C, respectively. For, the example and comparative example shown in Graphs 3 and 4, respectively, the growth experiment was conducted for one hour and the temperature of the crystallization area (second reaction zone) was set to 1000°C with HCl input partial pressure of  $1 \times 10^{-3}$  atm and NH<sub>3</sub> input partial pressures of 0.1 atm. Please note that alpha in Graphs 3 and 4 represents an absorption index. Thus, Graph 3 illustrates an absorption spectra of the AlN layer at the first reaction zone for the example having the Al material area set to 600°C, and Graph 4 illustrates an absorption spectra of the AlN layer at the first reaction zone for the comparative example having the Al material area set to 850°C.

The absorption spectra in Graph 3 has a sharper rising shape than that of the absorption spectra in Graph 4. In the data of Graph 4 (first reaction zone at 850°C), there are absorption spectra at the range of photon energy under the edge of the band ( $=5.818\text{eV}$ ), while the absorption spectra in Graph 3 (first reaction zone at 600°C) at the range of photon

energy under the edge of the band ( $=6.109\text{eV}$ ). Thus, the crystallinity of AlN of Graph 3 (at  $600^{\circ}\text{C}$ ) is higher than that of Graph 4 (at  $850^{\circ}\text{C}$ ). The crystallinity of AlN at  $600^{\circ}\text{C}$  is higher than the crystallinity of AlN at  $850^{\circ}\text{C}$  in the comparative example because the quartz reaction tube is not corroded at the temperature range of  $700^{\circ}\text{C}$  or below, such as  $600^{\circ}\text{C}$  as in the example of Graph 3.

Thus, the results illustrated in Graphs 1-4 of the present declaration demonstrate that reacting a solid Al with a halogenated hydrogen at a temperature of  $700^{\circ}\text{C}$  or below in a first reaction zone of the reaction chamber is critical to producing an Al component such as  $\text{AlCl}_3$  for transporting into the second reaction zone that prevents the quartz reaction tube of the second reaction zone from being corroded, damaged or discolored by the Al component therein.

6. I hereby declare that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine and/or imprisonment under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing therefrom.

Date: August 10, 2007



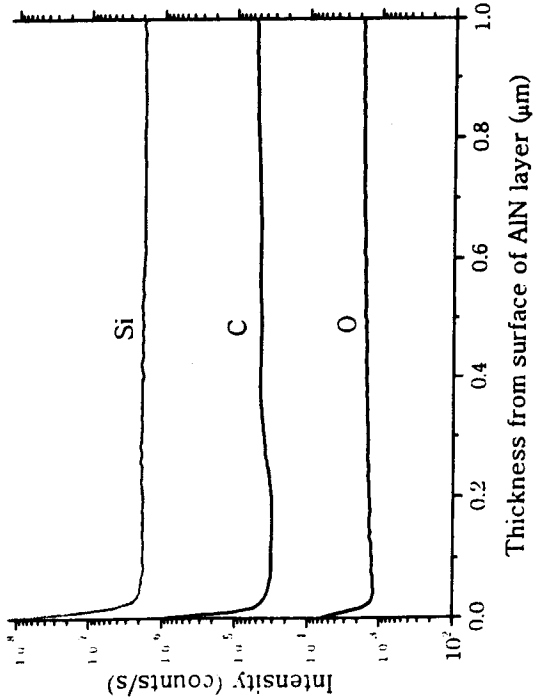
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Koukitu AKINORI

Attachments:  
Graphs 1-4

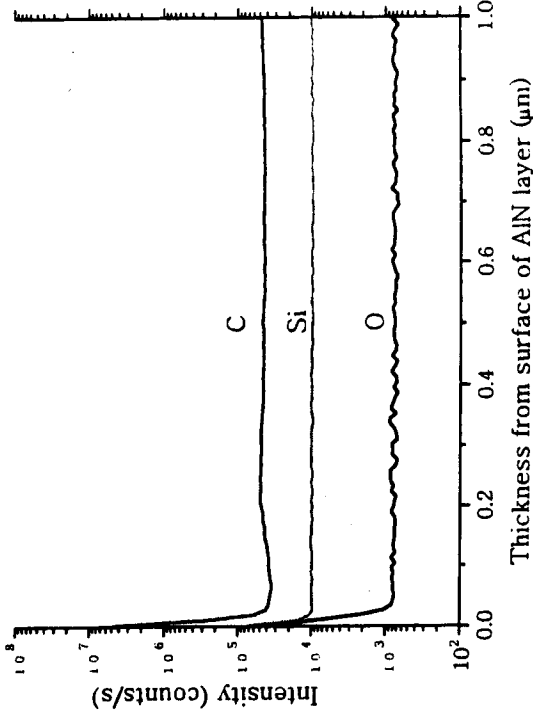
# Secondary Ion Mass Spectroscopy (SIMS) depth profile

Al source temperature: 850°C



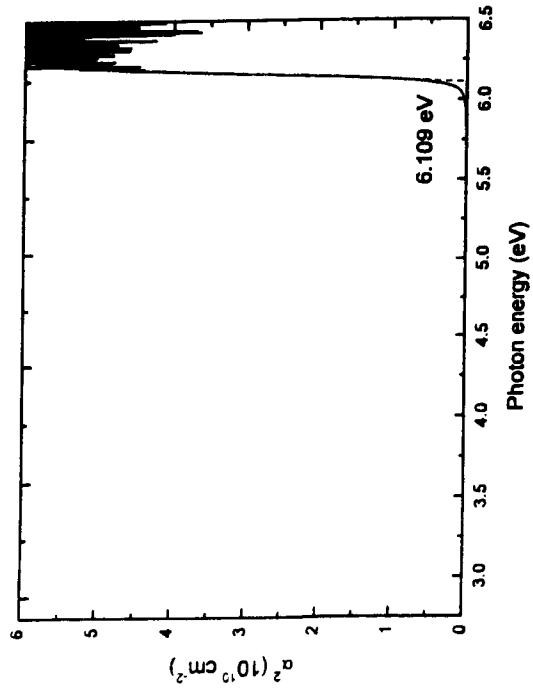
Graph 1

Al source temperature: 650°C

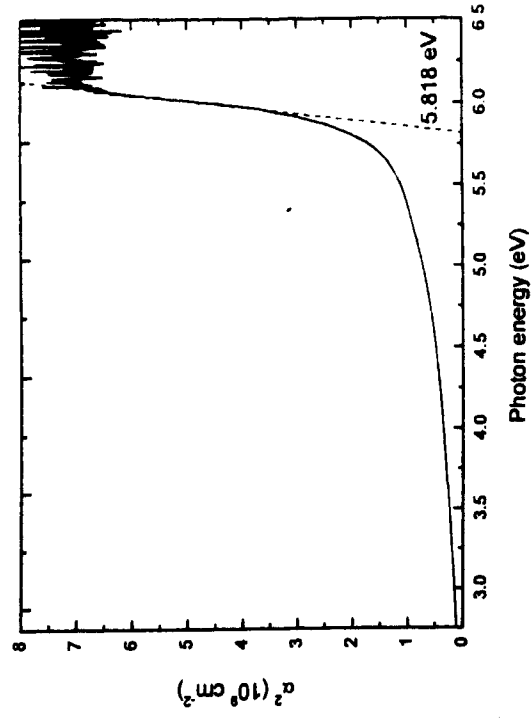


Graph 2

# Absorption spectra of AlN layer



Graph 3



Graph 4